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DESIGN, FABRICATE, AND DEMONSTRATE AN INSTRUMENT FOR ASSAYING BENZO (e) PYRENE

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DESIGN, FABRICATE, AND DEMONSTRATE AN INSTRUMENT FOR ASSAYING BENZO (a) PYRENE

by

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A. Introduction

1. This report and manual contains instructions for the installation, operation, and maintenance of a Hewlett-Packard 5700A Gas Chromatograph with electron capture detector (ECD) using a gas-phase stripper and injection system interfaced with an Aminco Bowman Spectrophotofluorometer detector (SPFD). Specific examples of usage and maintenance procedures as well as cautions are included.

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16. ABSTRACT

The purpose of this contract was to develop a method for the assay of Benzo(a)pyrene (BaP) directly from particulate matter.

A gas chromatograph with a gas phase fluorescence detector was used for the assay of the Benzo(a)pyrene.

The BaP along with many other compounds was vaporized from the particulate into a cartridge containing the same material that was in the analytical column of the gas chromatograph.

The cartridge was interfaced to the gas chromatograph where its contents could be heated and injected into the analytical column of the gas chromatograph. Although only partial separation of BaP is achieved in the analytical column, complete discrimination of BaP from other compounds is obtained by means of the gas phase fluorescent detector. This discrimination was obtained by choosing excitation and fluorescent emission wavelengths that were selective for Benzo(a)pyrene. This system can detect BaP at concentrations as low as 0.1 ug/1000 m^3 of air.

17. KEY WORDS AND DOCUMENT ANALYSIS

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3. Applications

1. This instrument package has been developed to allow the analysis of polynuclear arenes (PNA's) directly from particulate matter using a gas-phase high-temperature stripper and injector. It has been successfully applied to the analysis of many PNA's (including benzo[a]pyrene which is of main interest) directly from ground glass fiber filter samples of air particulate matter and leaded as well as unleaded automobile fuel exhaust particulates.

2. The SPF has been modified to accept the effluent from a gas chromatograph with ECD (see Fig. 1) which allows detection of PNA's by EC and SPF sequentially and practically simultaneously.

3. The instrument has been applied to the GLC analysis of standardized solutions of PNA compounds of interest. As shown in Figure 2, the fluorescence response to pyrene (50-200 ng), benzo [a]pyrene (100-400 ng), benzo[k] fluoranthene (20-80 ng), and chrysene (100-400 ng) is linear for standard syringe injected samples over the ranges shown.

4. Soxhlet (or sonified) solvent extracts of air particulates can be analyzed by syringe injection or gas phase injection. Figure 3 shows an example of a Louisville, Kentucky, air particulate sample compared to a PNA standard. The upper trace (SPF signal only) is from a 1 µl injection of the standard and the lower one

from 15 µl of a soxhlet extract of 1 gram of the fiberglass filter with unknown particulate loading. The solvent had been reduced to 1 ml total volume before the portion (15 µl) was removed for injection.

5. A gas phase injection of a New York City air particulate sample is shown in figure 4. A 100 mg ground filter sample was stripped of its PNA's using the high temperature stripper and injected via the gas phase injector onto the GLC column. The fluorescence signal is enhanced as compared to that of a soxhlet extracted sample due to the ability of injecting the total amount of PNA's in the sample rather than a small portion of a solvent extract while using only 1/10 as much filter sample.

C. Description of Instrument Package

1. An Aminco-Bowman SPF with a Xenon lamp source, ellipsoidal condensing mirror system, optical unit, photomultiplier microphotometer, power supply, and accessories.

2. A Hewlect Packard 5700A Gas Chromatograph equipped with a linear pulsed electron capture detector is interfaced with the SPF and contains a gas-phase injector system consisting of an eight-port high-temperature Valco valve and high-temperature asbestos insulated aluminum oven assembly which accepts 1/4" o.d. x 5" sample tubes.

3. A gas-phase stripper which consists of an asbestos insulated aluminum block with holes for accepting four 3/8" o.d. sample tubes and one 3/8" o.d. 250-watt heater cartridge and a gas inlet manifold with connecting hardware.

4. Four variable voltage transformers.

5. Model 7128A Hewlett Packard Strip Chart Recorder with dual pens.

6. A Thermo-Electric 6-station pyrometer.

D. Installation and Initial Turn-On

1. ECD

a. Refer to Initial Turn-On (pages 2-17) in 5700A G. C. Operating Manual.

b. After leak testing the column and flow system (pages 2-18, #5), adjust the 5-10% Methane in Argon carrier gas pressure to 50 psi and rotate the carrier flow control knobs A and B counterclockwise until maximum flow is obtained.

NOTE: Only metal columns (stainless steel with brass ferrules and nuts) should be used at carrier gas pressure above 50 psi.

c. Turn the ECD temperature control to 350°C and the injector port control to 300°C, and allow to reach equilibrium before turning the column temperature to desired level (see section on columns in H.P. manual)

d. Before interfacing the ECD to the SPFD check the flow rate from the ECD exit port to determine actual value for later comparison after interfacing of SPFD.

2. Gas-Phase Injector

a. The temperature control for the 8-port valve connecting the injector to the column, marked auxiliary temperature on the 5708A control panel, should be set at 300°C.

NOTE: The valve should not be operated above the 300°C auxiliary temperature setting or damage to the seals may result.

b. The temperature control for the sample chamber of the injector is controlled by a 120-140 V transformer independent of the GC and should be turned off until samples are ready to be run.

c. The 8-port valve stem should be turned counterclockwise to allow carrier gas to flow through the closed loop and into the analytical column (see Fig. 5A). The clockwise position is used only for gas-phase sample injection (Fig. 5B).

NOTE: Never leave the heated valve in an intermediate position, as this stops the flow to the ECD.

3. SPFD

a. The Xenon lamp is ignited by turning the power switch "on" and then pushing the "starter" button.

b. The focus of the Ellipsoidal Condensing Mirror System has to be checked and correctly set in order to attain highest sensitivity. The small lid on the Xenon lamp housing (just above the blower hose) must be removed and with a mirror one can see the variable slit (0-5 mm calibrated thumb wheel). The slit should be closed and

light be directed in a small dot at the center of the slit doors. The focusing is accomplished by the large metallic and two smaller black thumb wheels. The small one to the left moves the light focus vertically, the other small one (on top) moves it horizontally and the large thumb wheel (in front) concentrates the light to a small dot of high intensity.

NOTE: If an RF-type power supply is used the GC must be unplugged and the left top lid of the GC removed and the electrometer leads disconnected before igniting the lamp.

c. Turn the meter multiplier (MM) power to the HV (High Voltage) position, making sure there are no light leaks which may damage the phototube. Allow ten minutes for the MM to equilibrate.

d. Turn on the recorder - the Channel 1 (blue pen) is the ECD and the Channel 2 (red pen) records the SPFD response, a 20-unit relative intensity (10 millivolt signal) should give a full-scale (100-unit) response on this recorder.

4. Interfacing the ECD and SPFD

a. The 1/8" tubing on the right side of the ECD is joined by an 1/8" to 1/8" S.S. Swagelok union to an ∿ 2' x 1/8" o.d. thin-wall S.S. tubing which serves as the transfer

line. This 1/8" tubing is bent in order to accommodate a connection to the underside of the SPFD cell compartment. The quartz flow cell is sealed at the bottom to a drilled-out 1/8" to 1/8" fitting with 2 silicone O rings to which the 1/8" tubing is connected with regular S.S. ferrules. The 1/8" tube which connects the ECD-SPFD must be heated by a heating element which is well insulated with fiber glass insulation. A thermocouple taped to the tubing allows monitoring of temperature of the line.

NOTE: The heating element consists of a 2' piece of asbestos cord with a tight coil of ∿ 25' (1.63 ohms/ft. at 20°C--∿ 45 Ω total) of Chromel "A" yellow 24-gauge (asbestos-covered) wire wound around it with an electrical plug-in joined to the ends of the wire coil and insulated by 2' of woven tubular fiberglass.

E. Operation

1. Sample Preparation

a. A fiberglass filter mat, whose total weight and particulate loading is known, is dry-ground in a Wiley mill to 20 mesh.

b. The ground filter mat is then thoroughly mixed and the desired number of portions of sample are removed for subsequent stripping. These portions should be accurately weighed for later quantitation of the PNA's and near 100 mg in size although considerably more or less may be needed depending on the PNA content of the particulate matter and its abundance on the mat.

c. The individual portions to be analyzed are inserted in 6" x 9 mm glass tubes and held in place with a tight plug of fiberglass wool at each end of the chopped sample.

d. These tubes are then placed in the stripper block and the appropriate 3/8" x 1/4" reducing unions are attached to the exposed ends of each tube using a back ferrule and two 1/4" silicone O-rings to seal them (stretching required to fit the 3/8" tubes). One end of each tube is connected to the stripper gas inlet manifold. Into the other end of each of these tubes is inserted a 1/4" x 5" glass sample tube containing the collector material.

This is 110/120 Mesh Anakrom A coated 10% by wt. with Dexsil 300 GC and it occupies approximately 3/4 of the length of each 4-1/2"-5" tube allowing for a glass wool plug on each end. Slightly less than 1/2 of the length of these tubes should protrude from the 1/4" fitting when installed in the stripper.

e. A flow of about 70 ml/min. of an inert gas is passed through each tube (some variations may be noted but due to the long stripping time, small differences in flow have negligible effect on recovery of PNA's from the particulate matter). The stripper block is then heated to 320-340° (for a 250-watt heater cartridge this requires ∿ 56% on a variac in the 140-V mode) and left operating 10-16 hours.

f. The sample tubes are removed after collection of the PNA's is complete and stored until used.

NOTE: The long-term stability of PNA's collected on these sample tubes had not been investigated; therefore, long storage times should be avoided if possible (> 2 weeks).

2. Gas-Phase Injection

a. The sample tube containing the PNA's and perhaps other condensed substances is inserted horizontally into the center of the coiled 1/16" tubing from left to

right and a 1/4" nut, reversed back ferrule and two silicone O rings are slipped onto each end. The carrier gas flows from left to right in the tube and should be in the same direction as the flow in the stripper. The tube is purged with an inert gas for 4-5 minutes to remove all traces of air and low boiling compounds. During this step, the valve stem should be in the counter-clockwise position, (Fig. 5A) thus allowing carrier gas to flow through a closed loop and into the analytical gc column. The purge gas enters the sample tube from a separate outside source connected to the 1/16" to 1/8" connector protruding from top of the valve.

b. After purging, a cap is put on the purge exit line (1/16" line next to the purge inlet), and the purge gas turned off from the tank. With the sample tube now free from air and other components which might harm the ECD, the condensed PNA's are now ready to be vaporized and injected into the analytical gc column.

c. The sample tube chamber lid is put into place, and the heater element in the sample chamber block turned on to full heat. The variable transformer controlling the 250-watt heater cartridge (3/8" x 3") positioned

lengthwise in the block is put in the 140-V position at full voltage (100%). The initial temperature of the sample chamber block is usually between 100-130°C due to heat transferred from the valve block. In approximately two minutes, the block temperature will have risen to 200°C at which point the valve stem is turned clockwise passing the carrier gas through the sample tube and into the gc column (Fig. 5B). This is marked on the chromatogram as the injection point from which the retention time for the various components can be measured. The chamber is allowed to heat up to 440°C which takes approximately six minutes from the injection point. CAUTION: DO NOT ALLOW THIS TEMPERATURE TO EXCEED 450°C. The heat is then turned off and the chamber allowed to cool slowly with the lid on for five minutes. The valve is turned at this time to divert carrier gas through the closed loop and the sample chamber opened to allow rapid cooling so that another sample can be readied for injection. The injection time is therefore thirteen minutes including an eight-minute heat-up and a five-minute hold time.

d. After the removal of the injected sample, insertion of the new one, and sample purge all can be accomplished while the previous sample is being chromatographed. Therefore, with the additional time of approximately 45 minutes needed to elute the sample, a nominal one hour per chromatogram is usual.

F. Maintenance

1. Gas chromatograph

Refer to Section 3, Routine Maintenance, pages 3-20 in the 5700-A GC Manual.

2. ECD

a. To clean the ECD, disconnect the gc column and the interfacing line from the inlet and exit, respectively, of the ECD. Turn the detector temperature down to 150°C and connect an 1/8" i.d. Teflon tube to the exit tube of the ECD. Using a 5 cc glass syringe connected to the Teflon tube, inject 5-10 cc each of hexane, benzene, methanol, and acetone through the ECD and collect the washings in a beaker as they emerge from the 1/4" entrance fitting inside the gc oven.

CAUTION: RADIOACTIVE COMPOUNDS MAY BE WASHED FROM THE DETECTOR, SO CARE SHOULD BE TAKEN TO FLUSH THE WASHINGS DOWN A DRAIN WITH PLENTY OF WATER. AVOID SKIN CONTACT WITH THESE SOLUTIONS.

3. Recorder Pens

a. When a recorder pen(s) stops writing, first check the ink supply.

b. If the pen is clogged, a piece of wire should be introduced into the tip until the blockage is removed. Remove

the ink line from the back-end of the pen, and pass water through the pen, using the syringe and tubing supplied with the recorder until no resistance to flow can be felt. The ink line is then connected to the rear of the pen and the ink priming bulb is used to pump ink to the tip. Avoid recorder operation with high amplitude rapid noise as this tends to lead to pen clogging.

4. Gas-Phase Injector

   a. Valve

      An indication of a leak in the injector system is an abnormally high flow reading on the carrier gas flow-meters A and B, on the side of the gc, or low flow rate reading from the gc effluent. The most likely spot for a leak is the 8-port valve which, when cooled below 300°C (aux. temp. control) cannot be expected to hold 50 psi pressure. However, if this temperature drop has not occurred, a leak on the valve may still be present. Harsh valve turning may precede the leaking and leaving the valve unused for long periods of time may also contribute to leakage. In this case, the asbestos board insulation on the back of the valve and sample blocks must be removed by unscrewing the two small screws from the back and the large screw that

bolts the asbestos board to the top of the gc and swinging the insulation just enough to allow access to the back of the valve block. Then the nuts which put tension on the spring on the valve shaft must be tightened to increase the tension and thus stop the valve from leaking.

CAUTION: EXCESSIVE TIGHTENING MAY CAUSE SCARRING OR CRACKING OF THE HIGH-TEMPERATURE VALVE SEATS.

b. Fittings

The 1/16" to 1/16" Swagelok union connecting the valve effluent line to the gc inlet is not likely to leak, since it is held rigidly in the valve block by the asbestos insulation. However, it can be leak-tested by removing the asbestos insulation from the back of the valve as before, cooling the injector to room temperature, and squirting the fitting with snoop-leak detector.

c. Sample Tube

The O-rings on the 5" x 1/4" sample tube and the analytical column are always potential leak sites and leaks there can be remedied by either tightening the fittings or changing the new O-rings. Also, it should be noted that at 50 psi carrier pressure cooling of the gc oven will almost invariably cause the rubber O-ring seals to leak.

CAUTION: WHEN THE XENON LAMP IS IGNITED, THE COLUMN OVEN HEATER MAY GO OFF. IT MUST ALWAYS BE

CHECKED AFTER IGNITION AND IMMEDIATELY BE TURNED BACK ON OR A CHANGE OF O-RINGS MAY BE NECESSARY.

5. Transfer Line

   a. Leaks

      Although most of the gas pressure occurs at the head of the analytical column, the fittings connecting the line from the gc effluent with the SPF should also be leak-tested before being insulated.

   b. Heating Element

      Excessive heating up and cooling down of the transfer line increases the probability of burning out the heater coil and should be avoided if possible. When heating the line, the variac dial should be on zero voltage then the switch turned to the 140-V setting and the dial increased slowly to near 40-45% or until a 300-350°C temperature is reached. Doing this over a several-hour period guards against a voltage surge to the coil and possibility of shorting it out.

6. SPFD Cell

   a. Heater Block

      The 100-watt heater cartridge which heats the cell holder should be brought up to a 300-320°C temperature in the same manner as the transfer line. However, it should be done even more slowly due to the slower heating rate of the brass cell holder.

b. O-Rings

The 1/8" O-rings used to seal the quartz flow cell to the transfer line need not be changed often due to the relatively small amount of carrier gas pressure on them as compared to those of the analytical column. However, as is true for the column, a cooling down of the cell holder will probably cause the O-rings to leak and need changing. In this case, the transfer line must be cooled, disconnected, the cell removed from the fitting, and two new 1/8" O-rings (holes slightly enlarged with a cork borer) used.

CAUTION: THE QUARTZ FLOW CELL IS VERY DELICATE AND EXPENSIVE, SO CARE MUST BE TAKEN IN REMOVING IT FROM THE FITTING AND IN TIGHTENING UP ON THE O-RINGS.

c. Cleaning Flow Cell

The flow cell can usually be cleaned by washing with solvents such as hexane, methanol, acetone, water, and methylene chloride (which removes any Dexsil-300 or 400 liquid phase from the cell) and using a small cotton swab. If it is heavily stained inside, soaking overnight in an Aqua Regia solution may aid in removing most stains and foreign matter.

IMPORTANT: THE OPTICAL SURFACES OF THE CELL SHOULD NEVER BE HANDLED WITH THE FINGERS.

7. Monochromators

a. Windows

The excitation and emission monochromator windows located in front of the slide slits must be cleaned of dust particles from time to time. The slide slits must be removed and a flexible cotton swab with ethanol and/or methylene chloride can be used to wipe the dust from the windows. This procedure can greatly enhance the sensitivity of the SPFD.

b. Sensitivity and Selectivity

For a good discussion of the use of slit size and arrangement to increase or decrease sensitivity and/or selectivity for the ECS refer to IV Operation, Section G, page 15 of the SPF instruction manual 768-H.

8. Photomultiplier Tube (PMT)

a. Fatigue of PMT (desensitization)

The PMT should never be exposed to sunlight or room light while the microphotometer is in the HV (high voltage) position. The PMT shutter should be in the up (closed) position when the lid to the cell compartment is removed or the SPF is shut down so as to avoid accidental desensitization of the PMT.

b. Insulation

By wrapping with fiberglass padding, the PMT housing may be insulated to some degree from heat radiated by the hot transfer line to minimize dark current and, to some extent, the shot noise (VII, B., 3 and 4).

9. Xenon Lamp Power Supply

a. Xenon Lamp Installation

IMPORTANT: The positive (+) terminal of the power supply (long white cable) is connected to the top (+) electrode on the ECS housing, and the negative (-) terminal (short heavily insulated cable) is connected to the bottom (-) electrode on the housing. To change the Xenon lamp, the side plate of the ECS (with blower hose connection) must be removed, and the lamp gently removed from its clamps. The end marked "BOTTOM" on the new lamp is inserted into the lower clamp or (-) electrode and the top electrode of the lamp into the upper (+) clamp. The small bump on the Xenon lamp is always placed facing away from the ellipsoidal mirror.

CAUTION: THE XENON LAMP MADE OF FUSED QUARTZ CONTAINS A POSITIVE PRESSURE OF XENON GAS (~ 5 atm.) AND SHOULD ALWAYS BE CONSIDERED AN EXPLOSION HAZARD. ALSO, NEVER LEAVE THE XENON LAMP ON WITHOUT THE BLOWER BEING ON OR THE PROBABILITY OF THE LAMP EXPLODING IS INCREASED (WHEN HOT THE PRESSURE INCREASES TO ~ 20 atm.).

See Aminco manuals.

b. Cables

Avoid allowing the Xenon lamp power supply cables to directly contact either each other or anything metallic as arcing may occur due to the high voltages present.

10. Stripper

a. Heater Cartridge

The transformer controlling the 250 watt heater cartridge which heats the stripper must be calibrated to give a 320-340°C temperature overnight. The aluminum block takes approximately two hours to heat up; however, if the voltage is too high, the temperature may rise above 340°C.

CAUTION: AT TEMPERATURES EXCEEDING 450°C, ALUMINUM BEGINS TO SOFTEN.

b. Leaks

Before allowing the stripper to heat up, a leak check of the stripper manifold, as well as the silicone O-rings sealing the 3/8" and 1/4" sample tubes, must be made to ensure the flow of $N_2(g)$ to the sample collection tubes to be constant.

NOTE: The O-rings used on the stripper need not be replaced as often as those of the gas phase injector and the analytical column.

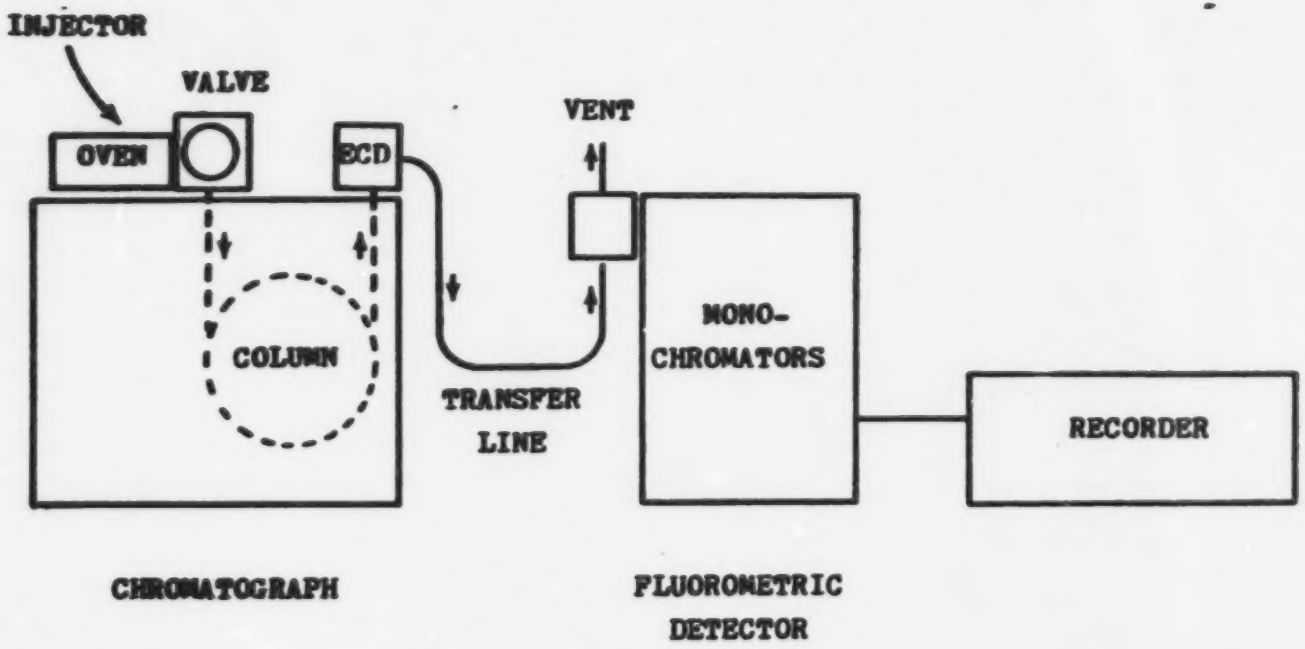

FIGURE 1

Schematic Diagram of ECD-SPFD
with Gas-Phase Injector

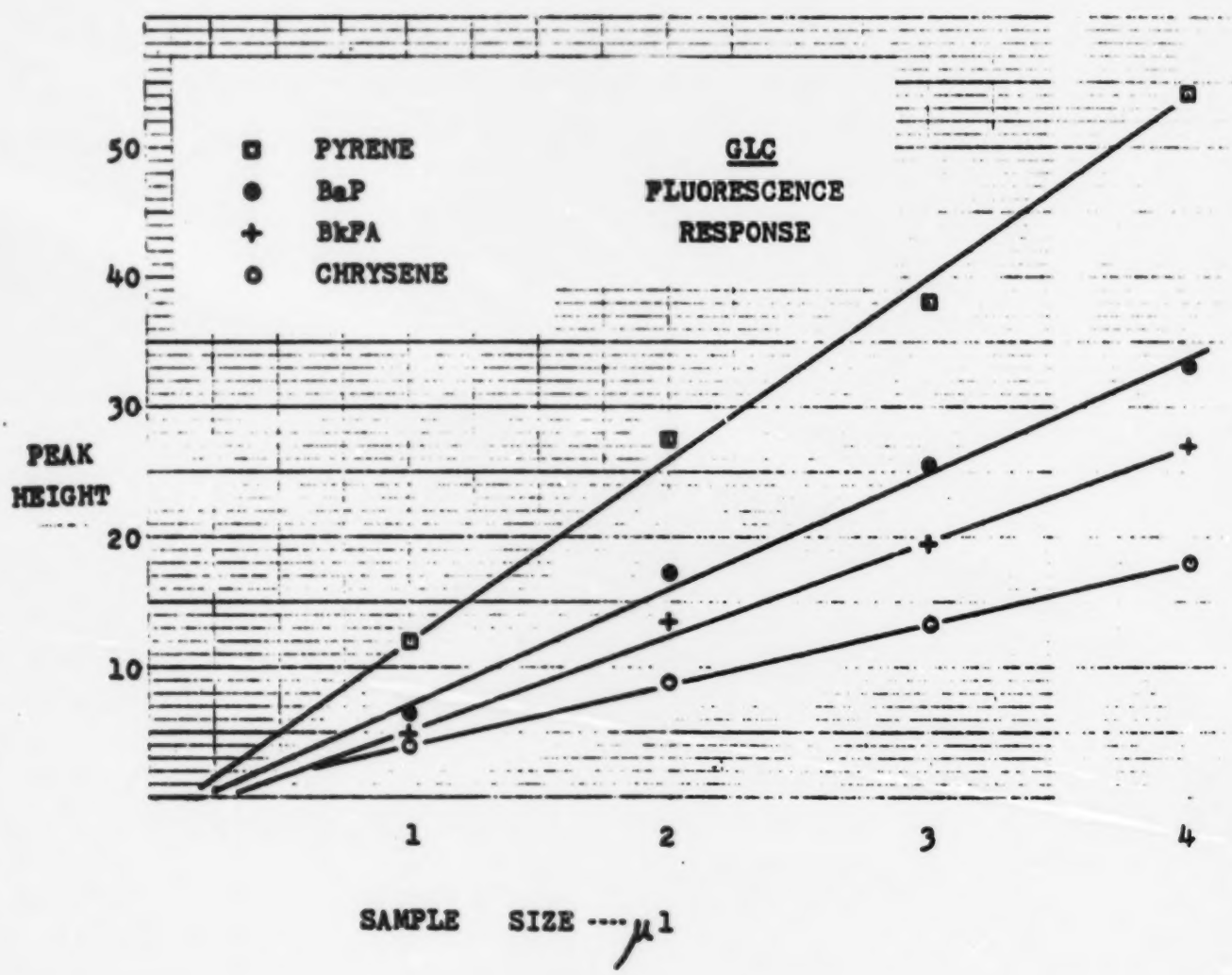

FIGURE 2

Linearity Plot

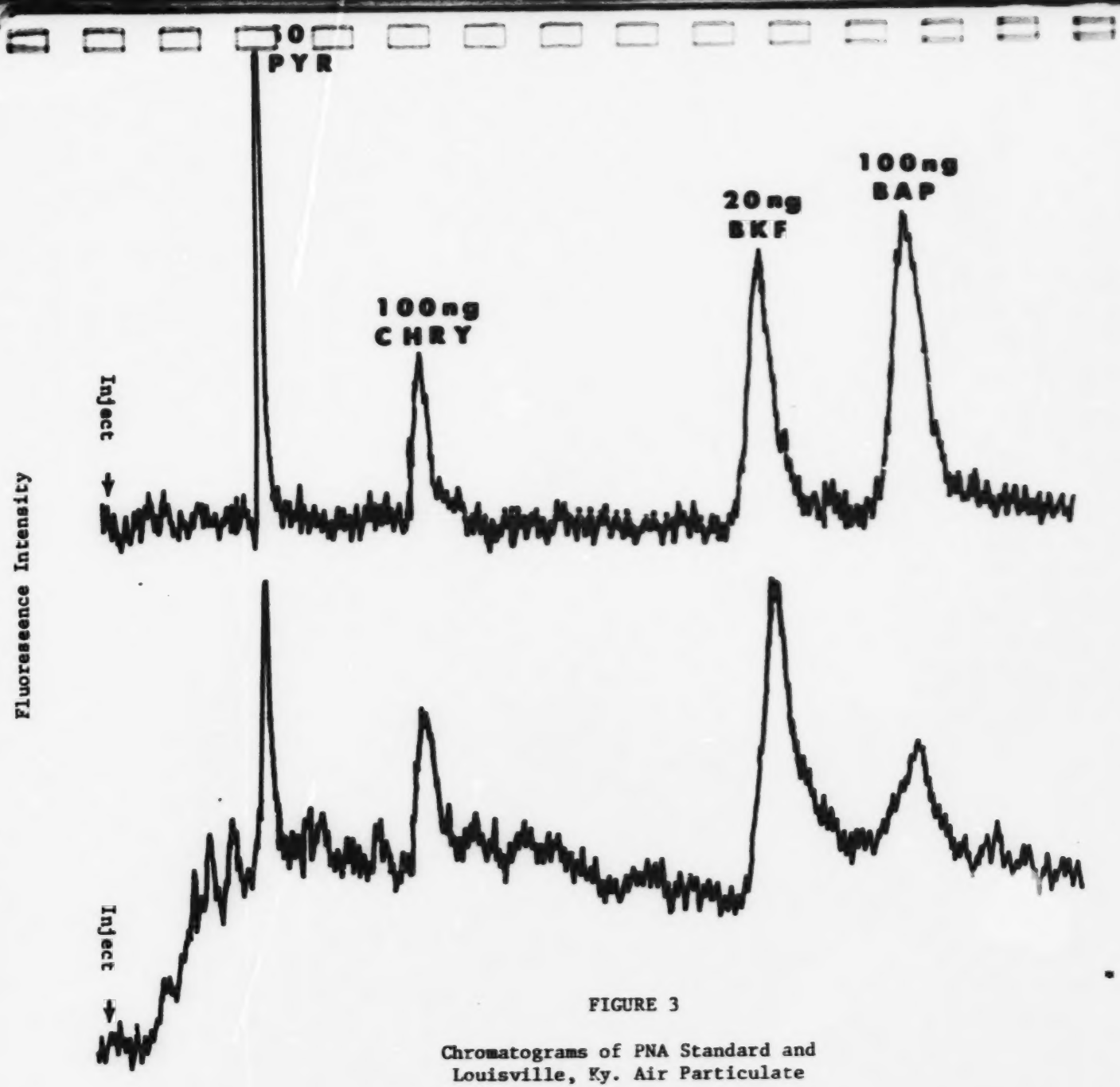

FIGURE 3

Chromatograms of PNA Standard and
Louisville, Ky. Air Particulate

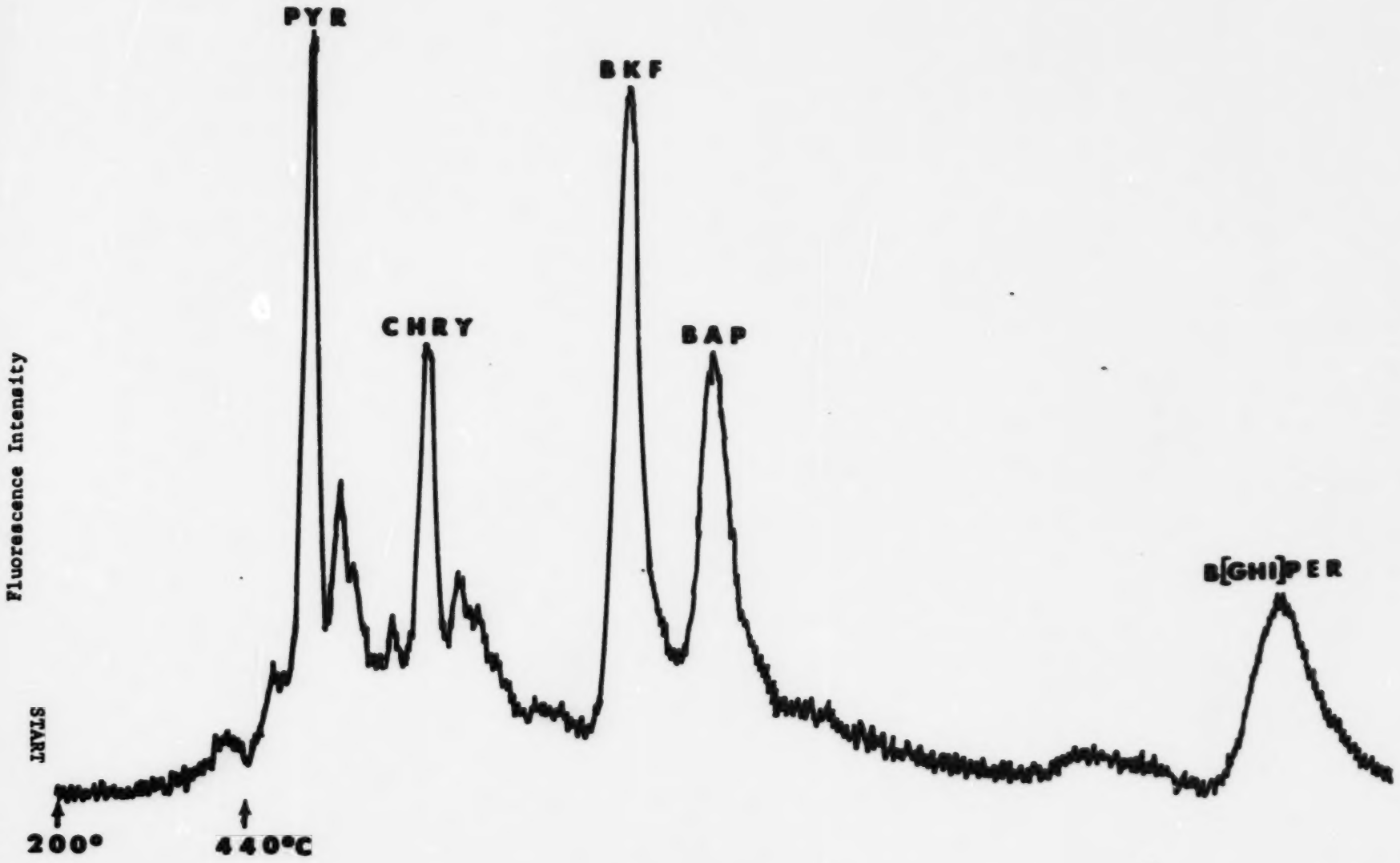

FIGURE 4

Gas-Phase Analysis of NYC Air Particulates

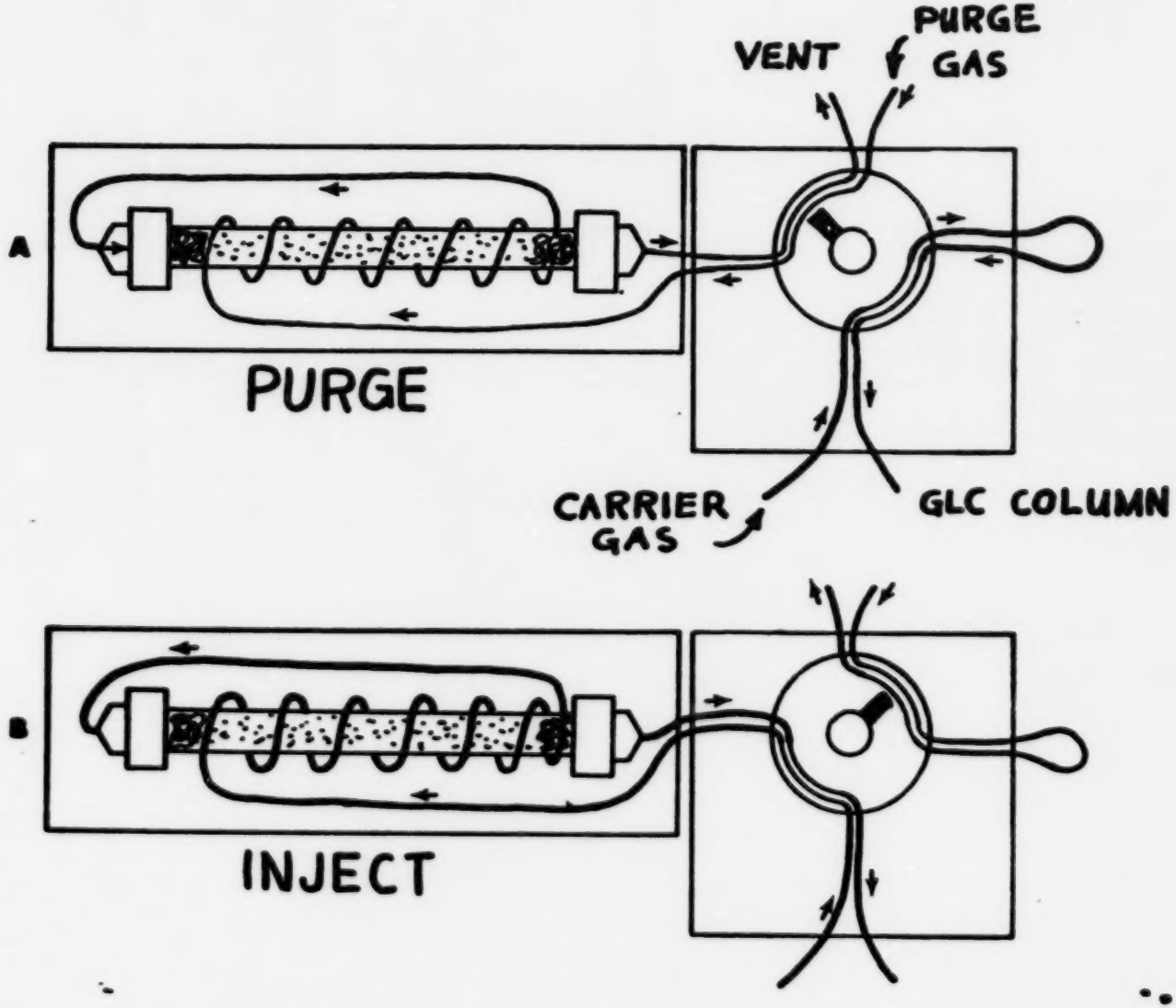

FIGURE 5

Schematic Diagram of Gas-Phase Injector